

MICROWAVE ASSISTED SYNTHESIS OF COPPER OXIDE NANOPARTICLES: OPTICAL AND STRUCTURAL CHARACTERIZATION

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ABSTRACT

Copper oxide (CuO) nanoparticles have been synthesized by precipitation method using copper nitrate and sodium hydroxide. The synthesized copper oxide nanoparticles have been characterized by using UV - Visible, FT-IR, XRD and TEM. XRD studies show that copper oxide has single phase crystalline monoclinic structure and average particle size is 23 nm. The TEM images show a homogeneous distribution of spherical CuO nanoparticles with particle size are in the range of 12-20 nm. UV-Visible absorption analysis indicates the presence of optical band gap in the material. FT-IR spectrum analysis shows only three vibrations occurring at around 424 cm⁻¹, 501 cm⁻¹ and 600 cm⁻¹ for CuO nanoparticles, which can be attributed to the vibrations of Cu-O, which confirms the formation of highly pure CuO nanoparticles.

Keyword : Copper oxide, Nanoparticles, Optical, Structural

1. INTRODUCTION :

In recent years, Nanotechnology has attracted many researchers from various fields like biotechnology, physics, chemistry, material sciences, engineering, medicine. The oxides of transition metals are an important class of semiconductors, which have applications in magnetic storage media, solar energy transformation, electronics and catalysis [1-9].

Among all metal oxide nanoparticles, copper oxide has gained the most interest because of its wide applications, such as in solar cell technology, field emission, magnetic storage media, lithium ion batteries, gas sensing, drug delivery, magnetic resonance imaging, and field emission devices. Very few methods of synthesis of copper oxide particles have been reported as compared to other oxides. CuO particles have been synthesized using different methods like sonochemical method [10], sol-gel technique [11], one-step solid state reaction method at room temperature [12], electrochemical method [13], thermal decomposition of precursors [14], co-implantation of metal and oxygen ions [15] and ultrasonic spray pyrolysis [16]. In the present study, we have synthesized and investigate size, morphology and optical properties of the pure CuO nanoparticles.

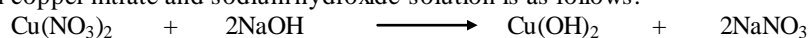
2. MATERIAL AND METHOD

2.1. Chemicals :

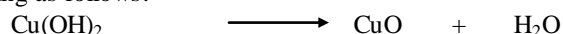
All chemicals used in the experiment are analytical reagent grade. Copper nitrate trihydrate [Cu(NO₃)₂.3H₂O] and Sodium hydroxide (NaOH) pellets were purchased from Merck, India. Deionised water was used throughout the experiment.

2.2. Synthesis:

In a typical synthesis procedure, 0.5 M sodium hydroxide solution is added dropwise with constant stirring to 0.5 M copper nitrate solution [Cu(NO₃)₂.3H₂O] until the pH of the system reaches to 12. The chemical reaction between copper nitrate and sodium hydroxide solution is as follows.



The resulting blue-green gel is washed with deionised water several times until free from nitrate ions. Finally the gel is dried by heating at 100⁰C for 10 hours on hot plate. Copper hydroxide decomposes into copper oxide on heating as follows.



The obtained product was kept at 500^oC for 5 hours in a muffle furnace to get the copper oxide nanocrystals. It was again activated in domestic microwave (900MW) for 30 minutes to get final copper oxide nanoparticles.

2.3. Characterization:

The average crystalline size of synthesized CuO was determined by X ray diffractogram obtained on X ray powder diffractometer. The FTIR of the sample was taken in the region between 400-4000 cm⁻¹. The UV-Visible absorption spectrum of CuO has been recorded by using double beam spectrophotometer. The morphology and particle size of the prepared CuO nanoparticles were obtained using transmission electron microscopy.

3. RESULT AND DISCUSSION

3.1. Structural Characterization ;

3.1.1. X-Ray Diffraction :

The XRD pattern of a prepared CuO nanoparticles as shown in fig. 1. It is well matched with the monoclinic phase of CuO (tenorite) nano particles. The lattice parameters are $a = 4.0160\text{\AA}$, $b = 3.4154\text{\AA}$, $c = 5.0276\text{\AA}$. The intensities and positions of peaks are in good agreement with the reported values (JCPDS file No. 05-661). No peaks of impurities are found in XRD pattern. The peaks are broad due to the nano-size effect. The average crystalline size of CuO nanoparticles is found to be 23 nm using Scherrer formula.

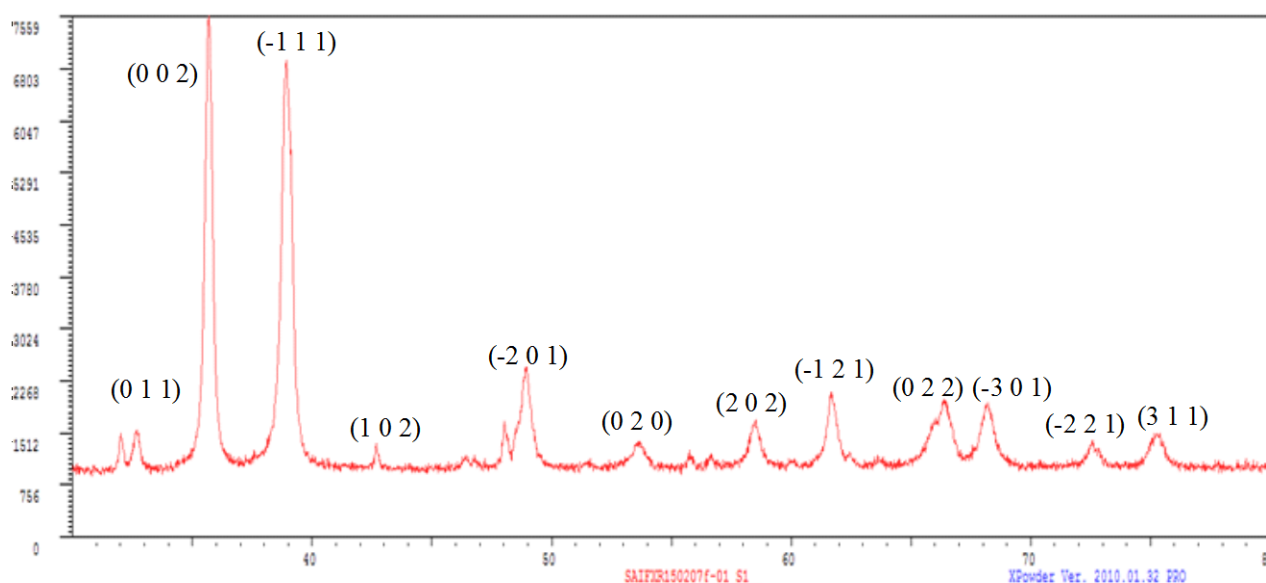


Fig-1. XRD pattern of CuO nanoparticles

3.1.2. Transmission Electron Microscope (TEM):

Figure-2 (a, b, c and d) shows the TEM images of prepared CuO nanoparticles. TEM images show a homogeneous distribution of spherical CuO nanoparticles. The size of particle observed in TEM images are in the range of 12-20 nm which is in good agreement with calculated by Scherrer formula using XRD.

3.2. Optical Properties:

3.2.1. UV-Visible Spectroscopy :

In order to explore the optical properties of CuO nanoparticles the optical absorption spectra was taken using UV-Visible spectrophotometer. Fig. 3 shows the UV-Visible absorption spectra of CuO nanoparticles annealed at 500^oC. The behavior obtained in UV-Visible spectrum can occur for variety of reasons, such as internal electrical fields within the crystal, deformation of lattice due to strain caused by imperfection and inelastic scattering of charge carries by photons [17]. From the UV spectra, it is clear that the absorbance decreases with increase in wavelength. This decrease in the absorption indicates the presence of optical band gap in the material.

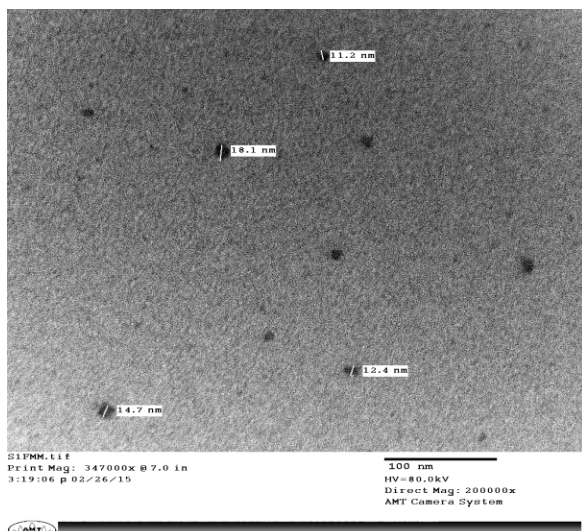


Fig-2 (a) TEM image

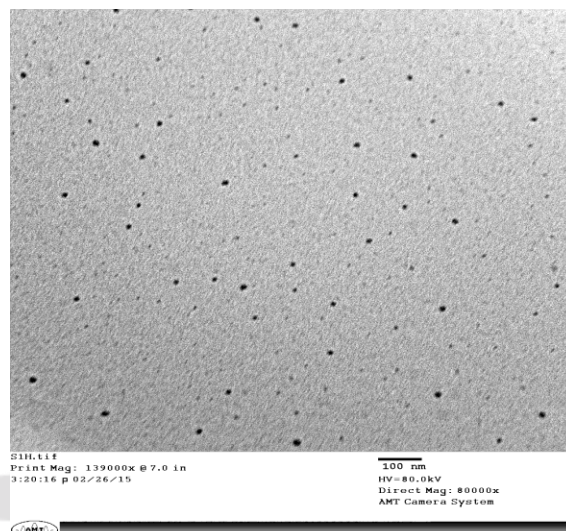


Fig-2 (b) TEM image

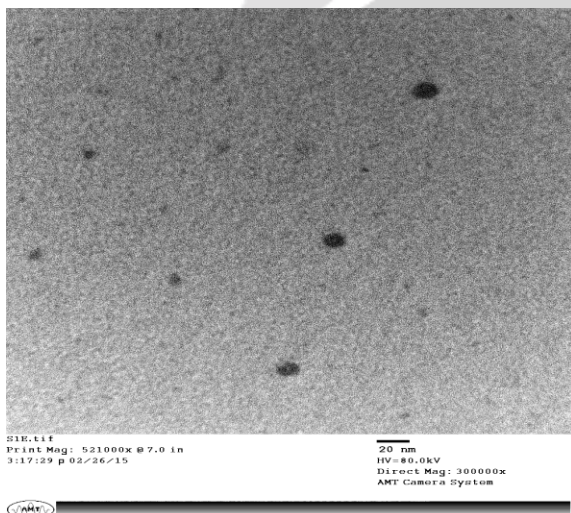


Fig-2 (c) TEM image



Fig-2 (d) TEM image

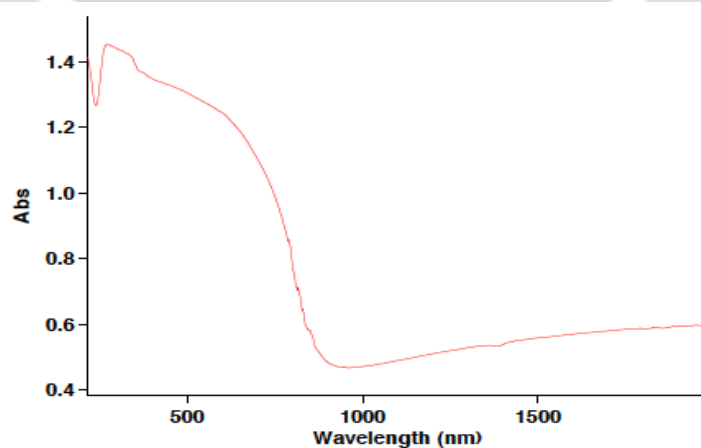


Fig.3. UV-Visible spectrum of CuO nanoparticles

3.2.2. FT-IR Spectroscopy :

FT-IR spectra were recorded in solid phase using KBr pellets technique in the region $3500\text{--}400\text{ cm}^{-1}$. FT-IR spectra of CuO nanoparticles treated at 500°C is shown in Fig. 4. FT-IR spectra exhibited in Fig. 4 shows only three vibrations occurring at around 424 cm^{-1} , 501 cm^{-1} and 600 cm^{-1} for all the samples which can be attributed to the vibrations of Cu-O, which confirms the formation of highly pure CuO nanoparticles. A weak band at around 2426 cm^{-1} may be attributed to the vibrations of atmospheric CO_2 . The present assignments agree well with the values reported in available literature [18,19,20].

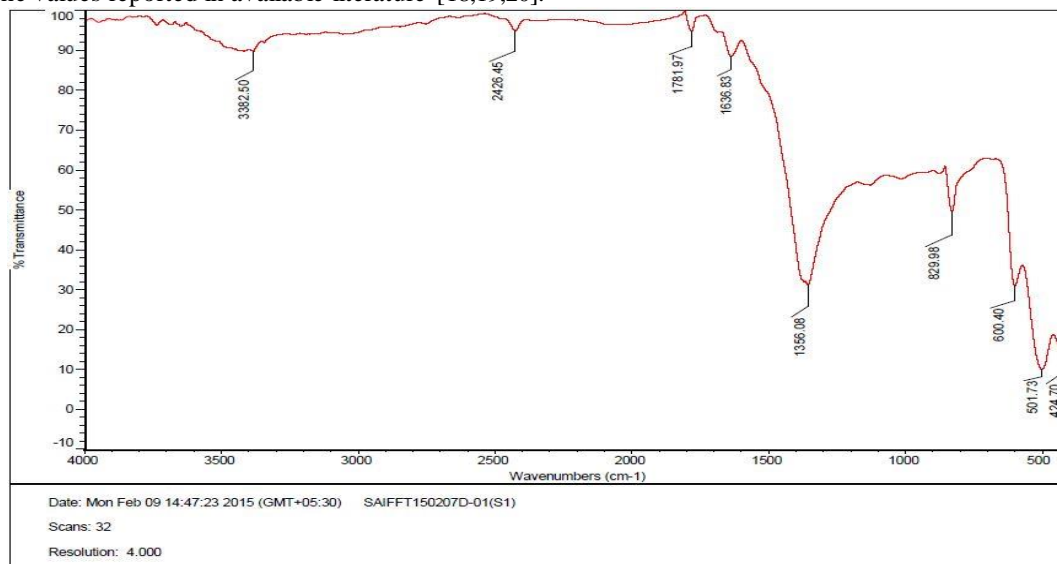


Fig. 4. FT-IR of CuO nanoparticles

4. CONCLUSION

The X ray diffraction analysis of CuO nanoparticles prepared in the present study shows CuO is crystalline with monoclinic structure and average particle size is 23 nm. The TEM images show a homogeneous distribution of spherical CuO nanoparticles with particle size are in the range of 12-20 nm. From UV-Visible absorption analysis the decrease in the absorption indicates the presence of optical band gap in the material. FT-IR spectrum analysis shows vibrations occurring at around 424 cm^{-1} , 501 cm^{-1} and 600 cm^{-1} for CuO nanoparticles, which can be attributed to the vibrations of Cu-O, which confirms the formation of highly pure CuO nanoparticles.

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